भारतीय मानक
Indian Standard

IS 1448 (Part 145) : 2022

पेट्रोलियम और इसके उत्पादों की परीक्षण पद्धतियाँ

भाग 145 परमाणु अवशोषण स्पेक्ट्रोस्कोपी द्वारा ईंधन तेल और कच्चे तेल में सोडियम,निक्कल और वैनेडियम का निर्धारण

(पहला पुनरीक्षण)

Methods of Test for Petroleum and its Products

Part 145 Determination of Sodium, Nickel and Vanadium in Fuel Oils and Crude Oils by Atomic Absorption Spectroscopy

(First Revision)

ICS 75.080

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December 2022 **Price Group 6**

Methods for Sampling and Test for Petroleum and Related Products of Natural or Synthetic Origin (excluding Bitumen and Hydrogen) Sectional Committee, PCD 01

FOREWORD

This Indian Standard (Part 145) (First Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Methods for Sampling and Test for Petroleum and Related Products of Natural or Synthetic Origin (excluding Bitumen and Hydrogen) Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This standard prescribes a test method for the determination of sodium and nickel at levels above 1 mg/kg and vanadium at levels above 10 mg/kg in crude oils and residual fuel oils. When fuels are combusted, metals present in the fuels can form low melting compounds that are corrosive to metal parts. Metals present at trace levels in petroleum can deactivate catalysts during processing. These test methods provide mean of quantitatively determining the concentrations of vanadium, nickel and sodium. Thus, these test methods can be used to aid in determining the quality and value of the crude oil and residual oil.

This standard was originally published in 1994 and in this first revision, the scope has been extended to analyse the samples producing particles while dissolving in organic solvents.

In the formulation of this standard, assistance has been derived from ASTM D5863-00a ‘Standard test methods for determination of nickel, vanadium, iron, and sodium in crude oils and residual fuels by flame atomic absorption spectrometry’.

In this revision precision data was provided by IOC (R&D).

The composition of the Committee responsible for the formulation of this standard is given in Annex A.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 2022 ‘Rules for rounding off numerical values (*second revision*)’.

Indian Standard

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

PART 145 DETERMINATION OF SODIUM, NICKEL AND VANADIUM IN FUEL OILS AND CRUDE OILS BY ATOMIC ABSORPTION SPECTROSCOPY

(*First Revision*)

1 SCOPE

1.1 This method is intended for the determination of sodium and nickel at levels above 1 mg/kg and vanadium at levels above 10 mg/kg in crude oils and residual fuel oils by Atomic Absorption Spectrometry (AAS).

1.2 Difficulty may be experienced in sodium determination with some samples due to the particulate nature of the sodium compounds present in it. It is recommended that an ashing technique should be used for such samples.

WARNING — This standard does not purport to address all of the safety-problems associated with its use. It is the responsibility of the user of this specification to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2 REFERENCES

The following standard contain provisions which, through reference in this text, constitutes provisions of this standard. At the time of publication the edition indicated was valid. All standards are subject to revision, and parties to agreements based on the standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below :

IS No.	*Title*
1070 : 1992	Reagent grade water — Specification (*third revision*)

3 OUTLINE OF THE METHOD

3.1 Two methods have been described to determine sodium nickel and vanadium by AAS:

3.1.1 *Test Method A*

In this test method, flame AAS is used to analyze a sample diluted with an organic solvent for the determination of nickel, vanadium and sodium. The samples are diluted in an organic solvent, the solutions are aspirated into the flame of an atomic absorption spectrometer, and the absorption of the visible or ultra-violet resonance radiation of the element being determined is measured. For sodium and nickel, an acetylene/air flame is used and for vanadium, an acetylene/nitrous oxide flame is used. Calibration curves are established from measurements on standard solutions of metallo-organic compounds in the same solvent.

3.1.2 *Test Method B*

In this test method, flame AAS is used to analyze a sample decomposed with acid for the determination of nickel and vanadium. 1 to 20 g of sample are weighed into a beaker and decomposed with concentrated sulphuric acid by heating at 525 °C in a muffle furnace. Dilute nitric acid is used to digest the inorganic residue and the inorganic residue is evaporated to incipient dryness, dissolved in dilute nitric acid and made up to volume with dilute nitric acid. Interference suppressant is further added to the dilute nitric acid solution. For sodium and nickel, an acetylene/air flame is used and for vanadium, an acetylene/nitrous oxide flame is used. The instrument is calibrated with matrix-matched standard solutions. The absorption intensities measured are related to concentrations by the appropriate use of calibration data.

4 PURITY OF REAGENTS

4.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be used in this test method.

NOTE — ‘Pure chemical’ shall mean chemicals that do not contain impurities which affect the results of analysis.

5 TEST METHOD A — FLAME ATOMIC ABSORPTION WITH AN ORGANIC SOLVENT TEST SOLUTION

5.1 Apparatus

5.1.1 *Atomic Absorption Spectrometer*

Instrument shall be complete with hollow cathode lamps and burners with gas supplies to support air-acetylene and nitrous oxide-acetylene flames.

WARNING — Hazardous. Potentially toxic and explosive. Refer to the manufacturer’s instrument manual for associated safety hazardous.

5.1.2 *Test Solution Containers*

Glass or plastic vials or bottles, with screw caps and a capacity of between 50 ml to 100 ml. Glass bottles of 100 ml capacity are satisfactory.

5.2 Reagents

5.2.1 *Dilution Solvent*

Mixed xylenes, o-xylene, tetralin and mixed paraffin-aromatic solvents shall be used.

WARNING — Combustible. Harmful vapours.

NOTE — Solvent purity can affect analytical accuracy when the sample contains low concentrations in the range of few mg/kg of the analytes.

5.2.2 *High Purity Mineral Oil*

5.2.3 *Organometallic Standards*

Multi element concentrations containing 100 mg/kg concentration of each element shall be used.

5.3 Preparation of Standards and Test Solutions

5.3.1 *Test Solution*

Take a container and weigh a portion of well-mixed sample in it, to get a sample concentration of either 5 percent (*m/m*) or 20 percent (*m/m*). Mix well. The element is analyzed with a test solution containing 5 percent (*m/m*) sample, if the concentration of sodium, nickel, or vanadium in the sample is more than 20 mg/kg. The analysis for the element with concentration less than 20 mg/kg is performed on a test solution that contains 20 percent (*m/m*) sample.

5.3.2 *Standards*

If the test solution contains 5 percent (*m/m*) sample, then the corresponding working standards and check standard must contain 5 percent (*m/m*) oil. Similarly, if the test solution contains 20 percent (*m/m*) sample, the standards must contain 20 percent (*m/m*) oil. A consistent dilution factor is vital so that all aspirated samples and standards will have the same viscosity. This is crucial to obtain consistent uptake rates.

5.3.3 *Working Standards*

Prepare a blank standard from mineral oil and three additional working standards from the organometallic standards that cover the ranges of concentration specified in Table 1.

Table 1 AAS Conditions for the Determination of Vanadium, Nickel, and Sodium following Solvent Dilution of the Sample

(*Clause* 5.3.3)

Sl No. (1)	Element (2)	Wavelength nm (3)	Concentration Range mg/kg (4)	Interference Suppressor (5)	Flame (6)
(i)	Vanadium	318.4[1]	0.5-15	15 mg/kg Al[2]	N_2O – C_2H_2
(ii)	Nickel	232.0[1]	0.5-20	None	Air-C_2H_2
(iii)	Sodium	589.0[1]	0.1-5	None	Air-C_2H_2

[1] or use appropriate wavelength (λ_{max}) as per the equipment manufacturer recommendation to obtain/get the required sensitivity/response in the target matrix/sample under testing

[2] prepared from an organometallic standard, mineral oil, and dilution solvent

5.3.4 *Check Standard*

Prepare a check standard to contain analyte concentrations approximately the same as expected in the test solution, using the organometallic standards, mineral oil, and dilution solvent.

5.4 Preparation of Apparatus

5.4.1 Refer to the manufacturer's instructions for the operation of the atomic absorption spectrometer. This test method assumes that good operating procedures are followed, as it is impractical to exactly specify required instrument settings due to design differences between spectrometers.

5.4.2 Set up the instrument to determine each analyte sequentially.

5.5 Calibration and Analysis

5.5.1 For each analyte in turn, perform the following operation :

5.5.2 Zero the instrument and nebulize the appropriate blank standard and working standards, determine the absorbance and establish a calibration curve of absorbance versus analyte concentration utilizing the instrument's concentration mode if available, otherwise plot these values. Determine if the calibration for each analyte is correct by using the check standard. Take corrective action and repeat the calibration, if the results obtained on the check standard are not within ±5 percent of the expected concentration for each analyte. Nebulize the test solutions. Measure and record the absorbance. Subtract the reagent blank absorbance to blank correct this absorbance, if appropriate.

5.5.3 Check the blank standard after measuring absorbance for a test solution. If this does not read zero, check the system, and then repeat **5.5.2**.

5.5.4 Test solutions showing absorbance greater than that obtained with the most concentrated working standard must be diluted. The dilution must contain interference suppressant at the specified concentrations.

6 TEST METHOD B — FLAME ATOMIC ABSORPTION AFTER ACID DECOMPOSITION OF THE SAMPLE

6.1 Apparatus — *see* **5.1**.

6.2 Reagents

6.2.1 *Aqueous Standard Solutions*

Aqueous standards prepared individually with concentration of 1 000 mg/kg for each vanadium and nickel purchased or prepared in acid matrix to ensure stability.

6.2.2 *Concentrated Nitric Acid*, HNO_3

WARNING — Poison, Oxidizer, Causes severe burns. Harmful or fatal if swallowed or inhaled.

6.2.3 *Nitric Acid* 50 *percent* (*v/v*) — Add one volume of concentrated nitric acid (*see* **6.2.2**) to one volume of water, stirring carefully.

6.2.4 *Dilute Nitric Acid*, 5 *percent* (*v/v*) — Add one volume of concentrated nitric acid (*see* **6.2.2**) to 19 volumes of water, stirring carefully.

6.2.5 *Concentrated Sulphuric Acid*, H_2SO_4

WARNING — Poison, Oxidizer, Causes severe burns. Harmful or fatal if swallowed or inhaled.

6.2.6 *Aluminum Nitrate*, $Al(NO_3)_3.9H_20$

6.2.7 *Potassium Nitrate*, KNO_3

6.3 Standard Solutions

6.3.1 *Multi-Element Standard*

Prepare a multi-element standard having 1 000 mg/kg each of vanadium and nickel with the aqueous standard solutions. Standards shall be prepared to ensure accuracy and stability and shall be stored in clean containers to prevent physical degradation.

6.3.2 *Working Standards*

Prepare at least two working standards covering the concentration ranges specified in Table 2. In case of vanadium, add the specified interference suppressant. Each working standard must contain 5 percent (*v/v*) nitric acid. Accuracy and stability shall be ensured while preparing the standards and shall be stored in clean containers to prevent physical degradation.

6.3.3 *Standard Blank*

Standard blank is a mixture of 5 percent (*v/v*) nitric acid and any interference suppressant specified in Table 2.

6.3.4 *Check Standard*

Prepare a calibration check standard in the same process as the working standards and at analyte concentrations that are typical of the specimens being analyzed.

Table 2 AAS Conditions for the Determination of Vanadium and Nickel Following Acid Sample Decomposition

(*Clauses* **6.3.3** and **6.4.8**)

Sl No.	**Element**	**Wavelength** nm	**Concentration Range** mg/kg	**Interference Suppressor**	**Flame**
(1)	(2)	(3)	(4)	(5)	(6)
(i)	Vanadium	318.4	0.5-20	250 mg/kg Al, $Al(NO_3)_3$ in 5 percent (*v/v*) HNO_3	N_2O – C_2H_2
(ii)	Nickel	232.0	0.5-29	None	Air-C_2H_2

6.4 Preparation of Test Solutions

6.4.1 Weigh an amount of sample estimated to contain between 0.002 5 mg and 0.12 mg of each metal to be determined in a beaker. Add 0.5 ml of H_2SO_4 per gram of sample.

> NOTE — To increase the lower concentration limits of the test method, the decomposition can be done in 10 g increments up to a maximum of 100 g. All the organic matter may not be needed to be destroyed each time before adding additional amounts of the sample and acid. To determine higher concentrations, sample size may be reduced accordingly.

6.4.2 Prepare reagent blanks using the equal amount of sulphuric acid as used for sample decomposition. Reagent blanks shall be carried through the same procedure as the samples.

WARNING — Reagent blanks are critical when determining concentrations below 1 mg/kg. Volume of acid and the dilutions as used for the samples, can also be used to simplify the analysis. For example, if 20 g of sample is being decomposed, use 10 ml of sulphuric acid for the reagent blank.

6.4.3 Place the beaker in the air bath, located in the hood. Ensure that the hot plate is off. Heat gently from the top with the infrared lamp and keep stirring the test solution with a glass rod. As decomposition begins (indicated by a frothing and foaming), control the heat of the infrared lamp to maintain steady evolution of fumes. Give equal and continuous attention to each sample mixture until all risk of spattering and foaming is over. Then, gradually increase the temperature of both the hot plate and lamp until the sample is reduced to a carbonaceous ash.

6.4.4 If the air bath apparatus is not used, heat the sample and acid on a temperature controlled hot plate. Keep an eye on the decomposition reaction and fine-tune the temperature of the hot plate accordingly.

WARNING — Hot fuming concentrated sulphuric acid is very corrosive and a strong oxidizing acid. Rubber gloves and a suitable face shield to protect against spattering acid, shall be used and the analyst shall also work in a well-ventilated hood.

6.4.5 Place the sample in the muffle furnace kept at 525 ± 25 °C. A gentle stream of oxygen may also be introduced into the furnace to expedite oxidation. Continue to heat until the carbon is completely removed.

6.4.6 Dissolve the inorganic residue by washing down the wall of the beaker with about 10 ml of the 50 percent (*v/v*) nitric acid. Digest on a steam bath for 15 min to 30 min. Transfer to a hot plate and gently evaporate to incipient dryness.

6.4.7 Wash down the wall of the beaker with about 10 ml of 5 percent (*v/v*) nitric acid. Digest on the steam bath until all salts are dissolved. Allow to cool. Transfer quantitatively to a volumetric flask of suitable volume and make up to volume with dilute nitric acid to get the test solution.

6.4.8 Pipette parts of the test solution into two separate volumetric flasks. Keep one flask for the determination of nickel. Add aluminum interference suppressant to the other flask for vanadium determination (*see* Table 2) and dilute up to mark with 5 percent (*v/v*) nitric acid. Similarly, prepare a reagent blank solution for vanadium analysis.

6.5 Preparation of Apparatus — *See* **5.4**.

6.6 Calibration and Analysis

6.6.1 For each analyte in turn, perform the following operation.

6.6.2 Zero the instrument and nebulize the appropriate blank standard and working standards, determine the absorbance and establish a calibration curve of absorbance versus analyte concentration utilizing the instrument's concentration mode if available, otherwise plot these values. Determine if the calibration for each analyte is correct by using the check standard. Take corrective action and repeat the calibration, if the results obtained on the check standard are not within ±5 percent of the expected concentration for each analyte. Nebulize the test solutions. Measure and record the absorbance. Subtract the reagent blank absorbance to blank correct this absorbance, if appropriate.

6.6.3 Check the blank standard after measuring absorbance for a test solution. If this does not read zero, check the system, and then repeat **6.6.2**.

6.6.4 Test solutions showing absorbance greater than that obtained with the most concentrated working standard must be diluted. The dilution must contain interference suppressant at the specified concentrations.

7 CALCULATION

7.1 For test Method A, calculate the concentration of each analyte in the sample using the following equation :

Analyte concentration, mg/kg

$$= C \times F$$

where

C = concentration of the analyte in the test solution, mg/kg; and
F = dilution factor, volume/volume or mass/mass.

7.2 For Test Method B, calculate the concentration of each analyte in the sample using the following using the following equation :

Analyte concentration, mg/kg

$$= (C \times V \times F)/W$$

where

C = concentration of the analyte in the test solution (corrected for the concentration determined in the reagent blank), µg/ml;

V = volume of the test solution, ml;
F = dilution factor, volume/volume or mass/mass; and
W = sample mass, g.

8 REPORT

Report the content of the appropriate metal to nearest mg/kg.

9 PRECISION

9.1 The following criteria shall be used for judging the acceptability of results (95 percent confidence):

9.1.1 *Repeatability, r*

Duplicate results by the same operator shall be considered suspect if they differ by more than the following amounts:

$r = 0.186\ X^{0.914}$ in the determination of sodium;
$r = 0.375\ X^{0.801}$ in the determination of nickel; and
$r = 0.150\ X^{0.976}$ in the determination of vanadium.

where, X is the mean of the two results, mg/kg.

9.1.2 *Reproducibility, R*

Results from two laboratories shall not be considered suspect unless they differ by more than the following amounts :

$R = 1.049\ X^{0.914}$ in the determination of sodium;
$R = 1.146\ X^{0.801}$ in the determination of nickel; and
$R = 0.585\ X^{0.976}$ in the determination of vanadium.

where, X is the mean of the two results, mg/kg.

NOTE — The estimates of R are based on very few degree of freedom and must therefore be regarded as tentative values.

9.1.3 The following are sample values of these expressions:

Sl No.	*Mean (in mg/kg)*	*Sodium*		*Nickel*		*Vanadium*	
		r	*R*	*r*	*R*	*r*	*R*
i)	10	1.5	8.6	2.4	7.3	1.4	5.5
ii)	20	2.9	16.2	4.1	12.6	2.8	10.9
iii)	30	4.2	23.5	5.7	17.5	4.2	16.2

Sl No.	*Mean* (*in mg/kg*)	*Sodium*		*Nickel*		*Vanadium*	
iv)	40	5.4	30.6	7.2	22.0	5.5	21.4
v)	50	6.6	37.5	8.6	26.3	6.8	26.6
vi)	60	7.9	44.3	10.0	30.4	8.2	31.8
vii)	70	9.0	51.0	11.3	34.4	9.5	37.0
viii)	80	10.2	57.6	12.5	38.3	10.8	42,1
ix)	90	11.4	64.1	13.8	42.1	12.1	47.3
x)	100	12.5	70.6	15.0	45.8	13.4	52.4

NOTE — It is anticipated that the above precision estimates can be improved as greater experience is gained of the method.

ANNEX A

(*Foreword*)

COMMITTEE COMPOSITION

Methods for Sampling and Test for Petroleum and Related Products of Natural or Synthetic Origin (excluding Bitumen and Hydrogen) Sectional Committee

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This Indian Standard has been developed from Doc No.:PCD 01 (18359).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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Published by BIS, New Delhi